National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1065b

Nickel Cyclohexanebutyrate

(Standard for Determination of Nickel in Petroleum Products)

This Standard Reference Material (SRM) is intended for use in the preparation of a standard of nickel in lubricating oils. SRM 1065b is essentially free from other metals and has suitable solubility, compatibility, and uniformity. It consists of a unit of 5 g of nickel cyclohexanebutyrate. The certified nickel content is given below.

Nickel . . . . . 13.89 ± 0.02 Wt %*

*Wt % = mg/kg x 10^{-4}

The uncertainty shown represents the 95% confidence limit of the mean based on fifteen deteminations and allowances for the effects of possible sources of known error.

Chemical Analysis Procedure: Nickel was determined by wet-ashing a 1 g sample (dried for 48 h over phosphorus pentoxide) with sulfuric and nitric acids, precipitating with dimethylglyoxime, and weighing the nickel dimethylglyoxime precipitate after drying at 150 °C. Determinations were also made using a solution prepared by wet-ashing a 0.1 g sample with nitric acid, fuming with perchloric acid, and dissolving the residue in water. The pH of an aliquot of this solution was adjusted to 13, treated with alcoholic dimethylglyoxime, and the nickel determined spectrophotometrically.

Spectrographic Analysis Procedure: The compound was examined spectrographically for metallic impurities. A 5 mg sample of the compound was excited in a direct-current arc and the photographed spectrum was examined for the characteristic lines of 51 elements. No significant impurities were found.

Stability: Tests show that standard lubricating-oil solutions of this compound with concentrations of nickel up to 500 ppm are stable for several weeks when prepared by the directions given on the reverse side.

Compatibility: Lubricating-oil solutions of this compound have been found to be compatible with lubricating-oil solutions of the other compounds in this series. Blends of several different compounds have been prepared by the procedures given in the certificates for the other metallo-organic compounds. However, tests have not been carried out to ensure compatibility with the various additives that may be in the oils to be analyzed.

Chemical analyses were performed by B.B. Bendigo, spectrophotometric analyses by E.R. Deardorff, and spectrographic analyses by V.C. Stewart. All analyses were performed at NIST, formerly NBS.

Technical and support aspects concerning the preparation, issuance, and update of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills.

Gaithersburg, MD 20899
November 3, 1993
(Revision of certificate dated 11-1-67)

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

(over)

Directions for Use: Transfer approximately 0.40 g of this compound from the bottle to a
fresh phosphorus pentoxide in a desiccator for 48 h. (Tightly close the bottle contain
compound.) Quickly and accurately transfer 0.360 g of this dried salt to a weighed 20
of salt is equivalent to 50 mg of nickel.) Add 3 mL of xylene and 5 mL of 2-ethylhexan
on a hot plate, with swirling and without charring, until a clear solution forms. Add to
mL of lubricating oil and gently shake the flask to mix the contents. Allow the flask to
and add enough lubricating oil to bring the total weight of the contents of the flask to 1
flask and shake gently to ensure a homogeneous solution. The concentration of nickel in

Source of Material: The nickel cyclohexanebutyrate was prepared by Distillation Produc
NY.